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Tris(1,10-phenanthroline)cobalt(II) bis(trichloroacetate)

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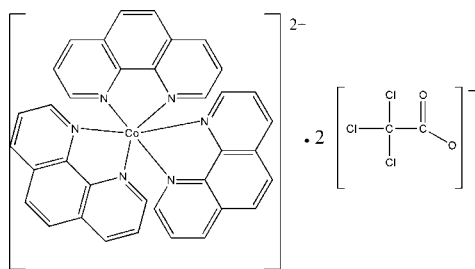
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.142; data-to-parameter ratio = 16.3.

In the title complex, $[\text{Co}(\text{C}_{12}\text{H}_8\text{N}_2)_3](\text{C}_2\text{Cl}_3\text{O}_2)_2$, the Co^{II} ion lies on a twofold rotation axis and is coordinated by six N atoms from three bis-chelating 1,10-phenanthroline ligands in a distorted octahedral environment. The crystal structure is stabilized by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For background to metal-organic framework coordination polymers, see: Chen *et al.* (2001); Fang *et al.* (2005). For a related structure, see: Harding *et al.* (2008).



Experimental

Crystal data

$[\text{Co}(\text{C}_{12}\text{H}_8\text{N}_2)_3](\text{C}_2\text{Cl}_3\text{O}_2)_2$
 $M_r = 924.28$
 Monoclinic, $C2/c$

$a = 18.367$ (4) Å
 $b = 10.753$ (2) Å
 $c = 19.020$ (4) Å

$\beta = 100.94$ (3)°
 $V = 3688.2$ (13) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.95$ mm⁻¹
 $T = 293$ K
 $0.26 \times 0.20 \times 0.12$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.837$, $T_{\text{max}} = 0.923$

17083 measured reflections
 4215 independent reflections
 3364 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.092$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.142$
 $S = 0.89$
 4215 reflections

258 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.81$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.45$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9A}\cdots\text{O1}^{\text{i}}$	0.93	2.54	3.401 (3)	154
$\text{C10}-\text{H10A}\cdots\text{O2}^{\text{ii}}$	0.93	2.35	3.120 (3)	140
$\text{C13}-\text{H13A}\cdots\text{O1}^{\text{iii}}$	0.93	2.28	3.004 (3)	134
$\text{C14}-\text{H14A}\cdots\text{O1}^{\text{iv}}$	0.93	2.60	3.455 (3)	154
$\text{C15}-\text{H15A}\cdots\text{O2}^{\text{iv}}$	0.93	2.56	3.266 (3)	133

Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (ii) $-x, -y, -z$; (iii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (iv) $x, -y + 1, z + \frac{1}{2}$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5253).

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supporting information

Acta Cryst. (2011). E67, m973 [doi:10.1107/S160053681102410X]

Tris(1,10-phenanthroline)cobalt(II) bis(trichloroacetate)

Li-Min Li, Yu-Feng Li, Li Liu and Zeng-Hui Zhang

S1. Comment

Metal-organic framework coordination polymers have attracted tremendous attention because of their molecular topologies and their potentially useful ionexchange, adsorption, catalytic and magnetic properties (Chen *et al.*, 2001; Fang *et al.*, 2005). As part of our search for new complexes of this type, we synthesized the title compound and report its crystal structure herein.

The molecular structure of the title complex is shown in Fig. 1. The Co^{II} ion lies on a twofold rotation axis and is coordinated by six N atoms of three bis-chelating 1,10-phenanthroline ligands in a distorted octahedral environment. The Co—N bond lengths are in agreement with those reported for a related complex (Harding *et al.*, 2008). The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds.

S2. Experimental

The title compound was obtained by adding 1,10-phenanthroline (3 mmol) dropwise to a solution of cobalt(II) trichloroacetic acid (1 mmol) in ethanol (20 ml). The solution was stirred for 1 h at room temperature. After a few days block-shaped crystals were formed from the yellow solution.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

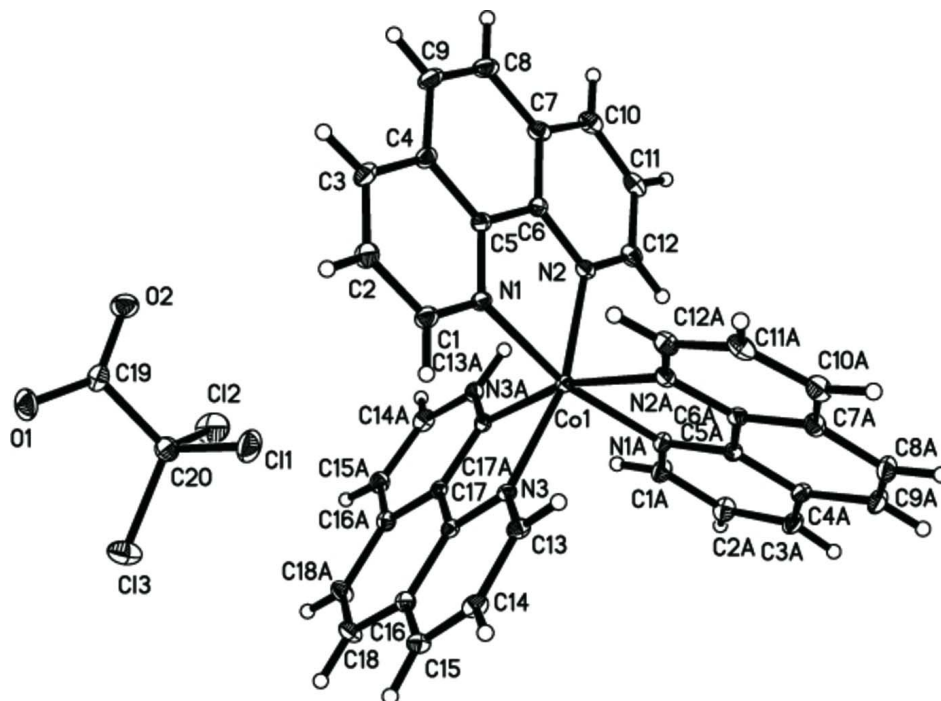


Figure 1

The molecular structure of the title compound showing 30% probability displacement ellipsoids (symmetry code: (A) $-x, y, -z + 1/2$). Only the unique anion is shown.

Tris(1,10-phenanthroline)cobalt(II) bis(trichloroacetate)

Crystal data

$[\text{Co}(\text{C}_{12}\text{H}_8\text{N}_2)_3](\text{C}_2\text{Cl}_3\text{O}_2)_2$

$M_r = 924.28$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 18.367\ (4)\ \text{\AA}$

$b = 10.753\ (2)\ \text{\AA}$

$c = 19.020\ (4)\ \text{\AA}$

$\beta = 100.94\ (3)^\circ$

$V = 3688.2\ (13)\ \text{\AA}^3$

$Z = 4$

$F(000) = 1868$

$D_x = 1.665\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3364 reflections

$\theta = 3.3\text{--}27.5^\circ$

$\mu = 0.95\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, yellow

$0.26 \times 0.20 \times 0.12\ \text{mm}$

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 9 pixels mm^{-1}

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.837, T_{\max} = 0.923$

17083 measured reflections

4215 independent reflections

3364 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.092$

$\theta_{\max} = 27.5^\circ, \theta_{\min} = 3.3^\circ$

$h = -23 \rightarrow 23$

$k = -13 \rightarrow 13$

$l = -22 \rightarrow 24$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.142$
 $S = 0.89$
 4215 reflections
 258 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.81 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.45 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.0000	0.08051 (4)	0.2500	0.01371 (15)
N1	0.08711 (11)	0.05924 (18)	0.19106 (10)	0.0166 (4)
N2	-0.04713 (11)	-0.05011 (17)	0.16840 (10)	0.0171 (4)
N3	0.05538 (10)	0.23619 (17)	0.30672 (9)	0.0139 (4)
C1	0.15367 (13)	0.1115 (2)	0.20262 (13)	0.0219 (5)
H1A	0.1671	0.1614	0.2429	0.026*
C2	0.20463 (14)	0.0959 (2)	0.15766 (14)	0.0244 (5)
H2A	0.2511	0.1330	0.1686	0.029*
C3	0.18537 (14)	0.0252 (2)	0.09727 (13)	0.0230 (5)
H3A	0.2174	0.0175	0.0651	0.028*
C4	0.11654 (13)	-0.0354 (2)	0.08449 (12)	0.0199 (5)
C5	0.06900 (12)	-0.0155 (2)	0.13273 (11)	0.0163 (4)
C6	-0.00237 (13)	-0.0750 (2)	0.12096 (12)	0.0158 (4)
C7	-0.02363 (13)	-0.1525 (2)	0.06160 (12)	0.0209 (5)
C8	0.02684 (15)	-0.1718 (2)	0.01384 (13)	0.0264 (5)
H8A	0.0134	-0.2243	-0.0253	0.032*
C9	0.09337 (15)	-0.1153 (3)	0.02451 (13)	0.0260 (5)
H9A	0.1248	-0.1286	-0.0077	0.031*
C10	-0.09434 (13)	-0.2060 (2)	0.05130 (13)	0.0234 (5)
H10A	-0.1101	-0.2593	0.0129	0.028*
C11	-0.13996 (13)	-0.1790 (2)	0.09837 (14)	0.0239 (5)
H11A	-0.1876	-0.2120	0.0918	0.029*
C12	-0.11401 (13)	-0.1013 (2)	0.15618 (13)	0.0209 (5)
H12A	-0.1453	-0.0843	0.1881	0.025*
C13	0.11155 (12)	0.2351 (2)	0.36144 (12)	0.0179 (5)

H13A	0.1308	0.1586	0.3786	0.022*
C14	0.14369 (13)	0.3426 (2)	0.39495 (12)	0.0208 (5)
H14A	0.1832	0.3371	0.4335	0.025*
C15	0.11645 (13)	0.4560 (2)	0.37044 (12)	0.0217 (5)
H15A	0.1360	0.5285	0.3931	0.026*
C16	0.05858 (12)	0.4615 (2)	0.31049 (12)	0.0173 (5)
C17	0.02949 (12)	0.3489 (2)	0.28041 (11)	0.0149 (4)
C18	0.02748 (14)	0.5754 (2)	0.27896 (14)	0.0225 (5)
H18A	0.0457	0.6508	0.2989	0.027*
C11	0.18800 (5)	0.41813 (7)	0.18548 (4)	0.0414 (2)
C12	0.06670 (4)	0.51120 (9)	0.07856 (5)	0.0454 (2)
C13	0.17181 (4)	0.67933 (6)	0.15928 (4)	0.0368 (2)
O1	0.25779 (11)	0.59450 (19)	0.05738 (11)	0.0341 (5)
O2	0.19590 (11)	0.41976 (17)	0.02240 (10)	0.0292 (4)
C20	0.16126 (14)	0.5325 (2)	0.11741 (13)	0.0245 (5)
C19	0.21116 (12)	0.5134 (2)	0.05881 (12)	0.0215 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0146 (2)	0.0140 (2)	0.0126 (2)	0.000	0.00274 (17)	0.000
N1	0.0169 (9)	0.0178 (9)	0.0155 (9)	-0.0009 (7)	0.0041 (8)	-0.0021 (7)
N2	0.0195 (9)	0.0134 (9)	0.0174 (9)	0.0003 (7)	0.0010 (8)	-0.0002 (7)
N3	0.0144 (8)	0.0157 (9)	0.0111 (8)	-0.0005 (7)	0.0013 (7)	-0.0017 (6)
C1	0.0217 (12)	0.0256 (12)	0.0189 (12)	-0.0045 (10)	0.0054 (10)	-0.0047 (9)
C2	0.0184 (11)	0.0283 (13)	0.0276 (13)	-0.0051 (10)	0.0071 (10)	-0.0035 (10)
C3	0.0237 (12)	0.0279 (13)	0.0198 (12)	0.0033 (10)	0.0101 (10)	0.0014 (9)
C4	0.0246 (12)	0.0207 (11)	0.0153 (11)	0.0041 (9)	0.0060 (9)	0.0025 (8)
C5	0.0186 (10)	0.0160 (10)	0.0140 (10)	0.0028 (9)	0.0025 (9)	0.0009 (8)
C6	0.0180 (10)	0.0154 (10)	0.0132 (10)	0.0015 (8)	0.0009 (9)	0.0018 (8)
C7	0.0260 (12)	0.0181 (11)	0.0164 (11)	0.0019 (9)	-0.0018 (10)	-0.0018 (8)
C8	0.0363 (14)	0.0254 (13)	0.0174 (12)	0.0043 (11)	0.0047 (11)	-0.0051 (9)
C9	0.0323 (13)	0.0300 (13)	0.0179 (12)	0.0043 (11)	0.0104 (11)	-0.0027 (10)
C10	0.0255 (11)	0.0203 (11)	0.0204 (12)	-0.0014 (10)	-0.0059 (10)	-0.0024 (9)
C11	0.0206 (11)	0.0164 (12)	0.0318 (14)	-0.0016 (9)	-0.0022 (10)	0.0017 (9)
C12	0.0176 (11)	0.0179 (11)	0.0264 (13)	-0.0005 (9)	0.0023 (10)	0.0006 (9)
C13	0.0184 (10)	0.0208 (11)	0.0138 (11)	0.0038 (9)	0.0009 (9)	-0.0003 (8)
C14	0.0160 (10)	0.0305 (13)	0.0135 (10)	-0.0014 (9)	-0.0033 (9)	-0.0025 (9)
C15	0.0248 (12)	0.0210 (12)	0.0191 (11)	-0.0033 (10)	0.0041 (10)	-0.0047 (9)
C16	0.0149 (10)	0.0187 (11)	0.0184 (11)	-0.0006 (9)	0.0037 (9)	-0.0009 (8)
C17	0.0147 (10)	0.0169 (11)	0.0140 (10)	0.0009 (8)	0.0053 (9)	-0.0002 (8)
C18	0.0270 (12)	0.0141 (11)	0.0247 (12)	-0.0015 (9)	0.0007 (10)	-0.0019 (9)
C11	0.0599 (5)	0.0325 (4)	0.0382 (4)	0.0076 (3)	0.0255 (4)	0.0144 (3)
C12	0.0190 (3)	0.0604 (5)	0.0581 (5)	-0.0049 (3)	0.0110 (3)	-0.0155 (4)
C13	0.0469 (4)	0.0273 (4)	0.0337 (4)	0.0072 (3)	0.0007 (3)	-0.0071 (3)
O1	0.0268 (10)	0.0393 (12)	0.0367 (11)	-0.0156 (8)	0.0074 (9)	-0.0009 (8)
O2	0.0271 (10)	0.0328 (10)	0.0277 (10)	-0.0036 (8)	0.0049 (8)	-0.0083 (7)
C20	0.0221 (11)	0.0250 (13)	0.0258 (13)	-0.0022 (10)	0.0033 (10)	-0.0008 (9)

C19	0.0148 (10)	0.0309 (13)	0.0176 (11)	-0.0020 (9)	0.0001 (9)	0.0034 (9)
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Geometric parameters (Å, °)

Co1—N1 ⁱ	2.1330 (19)	C8—H8A	0.9300
Co1—N1	2.1330 (19)	C9—H9A	0.9300
Co1—N3	2.1411 (18)	C10—C11	1.368 (4)
Co1—N3 ⁱ	2.1411 (18)	C10—H10A	0.9300
Co1—N2	2.1497 (19)	C11—C12	1.391 (3)
Co1—N2 ⁱ	2.1497 (19)	C11—H11A	0.9300
N1—C1	1.325 (3)	C12—H12A	0.9300
N1—C5	1.359 (3)	C13—C14	1.396 (3)
N2—C12	1.326 (3)	C13—H13A	0.9300
N2—C6	1.358 (3)	C14—C15	1.366 (3)
N3—C13	1.319 (3)	C14—H14A	0.9300
N3—C17	1.362 (3)	C15—C16	1.405 (3)
C1—C2	1.393 (3)	C15—H15A	0.9300
C1—H1A	0.9300	C16—C17	1.401 (3)
C2—C3	1.366 (4)	C16—C18	1.434 (3)
C2—H2A	0.9300	C17—C17 ⁱ	1.427 (4)
C3—C4	1.402 (3)	C18—C18 ⁱ	1.345 (5)
C3—H3A	0.9300	C18—H18A	0.9300
C4—C5	1.398 (3)	C11—C20	1.786 (3)
C4—C9	1.428 (3)	C12—C20	1.769 (3)
C5—C6	1.438 (3)	C13—C20	1.762 (3)
C6—C7	1.398 (3)	O1—C19	1.226 (3)
C7—C10	1.400 (3)	O2—C19	1.224 (3)
C7—C8	1.431 (3)	C20—C19	1.585 (3)
C8—C9	1.345 (4)		
N1 ⁱ —Co1—N1	167.69 (10)	C10—C7—C8	123.2 (2)
N1 ⁱ —Co1—N3	98.69 (7)	C9—C8—C7	121.2 (2)
N1—Co1—N3	90.95 (7)	C9—C8—H8A	119.4
N1 ⁱ —Co1—N3 ⁱ	90.95 (7)	C7—C8—H8A	119.4
N1—Co1—N3 ⁱ	98.69 (7)	C8—C9—C4	121.1 (2)
N3—Co1—N3 ⁱ	77.14 (10)	C8—C9—H9A	119.4
N1 ⁱ —Co1—N2	94.01 (8)	C4—C9—H9A	119.4
N1—Co1—N2	77.87 (7)	C11—C10—C7	119.2 (2)
N3—Co1—N2	164.22 (7)	C11—C10—H10A	120.4
N3 ⁱ —Co1—N2	93.40 (7)	C7—C10—H10A	120.4
N1 ⁱ —Co1—N2 ⁱ	77.87 (7)	C10—C11—C12	119.0 (2)
N1—Co1—N2 ⁱ	94.01 (8)	C10—C11—H11A	120.5
N3—Co1—N2 ⁱ	93.40 (7)	C12—C11—H11A	120.5
N3 ⁱ —Co1—N2 ⁱ	164.22 (7)	N2—C12—C11	123.7 (2)
N2—Co1—N2 ⁱ	98.40 (10)	N2—C12—H12A	118.1
C1—N1—C5	117.5 (2)	C11—C12—H12A	118.1
C1—N1—Co1	128.87 (16)	N3—C13—C14	123.5 (2)
C5—N1—Co1	113.61 (15)	N3—C13—H13A	118.2

C12—N2—C6	117.3 (2)	C14—C13—H13A	118.2
C12—N2—Co1	129.10 (17)	C15—C14—C13	119.2 (2)
C6—N2—Co1	113.39 (15)	C15—C14—H14A	120.4
C13—N3—C17	117.65 (19)	C13—C14—H14A	120.4
C13—N3—Co1	128.03 (16)	C14—C15—C16	119.1 (2)
C17—N3—Co1	114.28 (14)	C14—C15—H15A	120.4
N1—C1—C2	123.6 (2)	C16—C15—H15A	120.4
N1—C1—H1A	118.2	C17—C16—C15	117.8 (2)
C2—C1—H1A	118.2	C17—C16—C18	118.5 (2)
C3—C2—C1	119.1 (2)	C15—C16—C18	123.8 (2)
C3—C2—H2A	120.4	N3—C17—C16	122.7 (2)
C1—C2—H2A	120.4	N3—C17—C17 ⁱ	117.15 (12)
C2—C3—C4	119.0 (2)	C16—C17—C17 ⁱ	120.20 (13)
C2—C3—H3A	120.5	C18 ⁱ —C18—C16	121.32 (14)
C4—C3—H3A	120.5	C18 ⁱ —C18—H18A	119.3
C5—C4—C3	118.0 (2)	C16—C18—H18A	119.3
C5—C4—C9	119.2 (2)	C19—C20—C13	113.91 (17)
C3—C4—C9	122.7 (2)	C19—C20—C12	110.03 (16)
N1—C5—C4	122.6 (2)	C13—C20—C12	108.66 (14)
N1—C5—C6	117.7 (2)	C19—C20—C11	107.71 (17)
C4—C5—C6	119.6 (2)	C13—C20—C11	107.33 (13)
N2—C6—C7	122.9 (2)	C12—C20—C11	109.08 (14)
N2—C6—C5	117.3 (2)	O2—C19—O1	131.2 (2)
C7—C6—C5	119.9 (2)	O2—C19—C20	113.8 (2)
C6—C7—C10	117.9 (2)	O1—C19—C20	115.0 (2)
C6—C7—C8	118.9 (2)		

Symmetry code: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9A \cdots O1 ⁱⁱ	0.93	2.54	3.401 (3)	154
C10—H10A \cdots O2 ⁱⁱⁱ	0.93	2.35	3.120 (3)	140
C13—H13A \cdots O1 ^{iv}	0.93	2.28	3.004 (3)	134
C14—H14A \cdots O1 ^v	0.93	2.60	3.455 (3)	154
C15—H15A \cdots O2 ^v	0.93	2.56	3.266 (3)	133

Symmetry codes: (ii) $-x+1/2, -y+1/2, -z$; (iii) $-x, -y, -z$; (iv) $-x+1/2, y-1/2, -z+1/2$; (v) $x, -y+1, z+1/2$.